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IS 3025 (Part 57) (2005, Reaffirmed 2009): Method of Sampling and Test (Physical and Chemical) for Water and Wastewater, Part 57: Boron (First Revision). ICS 13.060.50

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IS 3025 (Part 57) : 2005

REAFFIRMED
2009

REAFFIRMED

भारतीय मानक

जल एवं अपशिष्ट जल के नमूने लेना और परीक्षण (भौतिक एवं रसायनिक) की विधियाँ

भाग 57 बोरान

(पहला पुनरीक्षण)

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 57 BORON

(First Revision)

ICS 13.060.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 2005 **Price Group 4**

Environment Protection and Waste Management Sectional Committee, CHD 32

FOREWORD

This Indian Standard (Part 57) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Boron is not found free in nature but is always bound to oxygen. It occurs generally as orthoboric acid and as alkali metal and alkaline earth metal borates. Boron constitutes only about 3 ppm of the earth's crust. The natural borate content of ground water, and surface water is usually small. The borate content of surface water can be significantly increased as a result of wastewater discharges, because borate compounds are ingredient of domestic working agents. Naturally occurring boron is present in ground water primarily as a result of leaking from rocks and soils containing borates and borosilicates. Concentration of boron in ground water throughout the world range widely < 023 to >100 mg/l. The majority of Earth's boron occurs in the oceans, with an average concentration of 4.5 mg/l. The amount of boron in fresh water depends on geochemical nature of the drainage area, proximity to marine coastal region and inputs from industrial and municipal effluents. As per IS 10500 : 1991 'Drinking water — Specification (*first revision*)', the permissible limits for boron in the drinking water is 1 mg/l, *Max*.

The Committee responsible for the formulation of IS 3025 : 1964 had decided to revise the standard and publish it in separate parts. This standard supersedes 29 of IS 3025 : 1964.

In the preparation of this standard considerable assistance has been derived from the following publications:

a) N. Howell Furman, Standard Methods of Chemical Analysis, Vol (I), D. Van Nostrand Company Inc. 1962. p. 209-238

b) Hayes M. R. and Metcalfe J., Analyst, 1962, 87 (956)

c) Harrison T. S and Coff W. D., Analyst, 1966, 91 (576)

d) Nemodruk A. A. and Karalova Z. K. Analytical Chemistry of Boron, Ann Arbor, London 1969

e) Kolthoff I. M. Elving P. J. and Robert S. Braman, Treatise on Analytical Chemistry P-II, Vol. XI, Interscience Publications

f) Sangita D. Kumar, B. Maiti and P. K. Mathur, Analytical Chemistry 1999, 71 (2551)

g) A. Tapparo, P. Pastore and G. G. Bombi, Analyst, 1998, 123 (1771)

Method prescribed in ISO 9390 : 1990 'Water quality — Determination of borate — Spectrophotometric method using azomethine — H' is applicable to potable water and to ground, surface and saline waters that are not heavily polluted. This method is incorporated in IS 13428 : 1998 'Packaged natural mineral water — Specification (*first revision*)'.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 57 BORON

(First Revision)

1 SCOPE

This standard (Part 57) describes the following five methods for the determination of boron in water and wastewater:

a) Titration method,
b) Colorimetric curcumin method,
c) Flow injection method,
d) Ion chromatographic method, and
e) Inductively coupled plasma atomic emission spectrometric method (ICPAES).

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and 7022 (Part 2) shall apply.

4 SAMPLE PRESERVATION

The sample bottles should be cleaned thoroughly with 6N HNO_3 prior to final rinsing with deionized water. The water samples should be collected and stored in polythene or silica containers. For preservation, the sample should be acidified with HCl (2 ml AR grade concentrated HCl in one litre of sample to bring down the *p*H below 2). For the analysis by flow injection or ion chromatographic method, the presentation time should be minimized and the acidification of the sample should be avoided. The sample should be filtered through 0.45 μ membrane filter before the analysis.

5 TITRATION METHOD

5.1 Principle

In acidic aqueous medium, boron in the solution is present as boric acid. Being a weak acid ($pH = 9.2$), it cannot be titrated satisfactorily against sodium hydroxide. However, if certain polyhydroxy compounds like mannitol are added, boric acid forms a strong monobasic acid complex, which can be titrated with a standard alkali, using phenolphthalein indicator.

5.2 Apparatus

5.2.1 *Glassware*, 250-ml beakers, glass rods, watch glass, 50-ml burette.

NOTE — Clean all glassware with potassium dichromate sulphuric acid cleaning solution.

5.3 Reagents

5.3.1 *Quality of Reagents*

Unless otherwise specified, pure chemicals and CO_2 free distilled water shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5.3.2 *Boric Acid Crystals*, AR Grade

5.3.3 *Mannitol*, AR Grade

5.3.4 *Sodium Hydroxide Solution* — 0.1 N (CO_2-free).

5.3.5 *Phenolphthalein Indicator* — 1 percent in 50 percent ethanol.

5.3.6 *Methyl Red Indicator* — 1 percent in 50 percent ethanol.

5.3.7 *Hydrochloric Acid* — 6 N.

5.4 Procedure

5.4.1 *Standardization of the Alkali*

Weigh accurately 100 mg of boric acid crystals in a 250-ml beaker and dissolve it in 200 ml distilled water. Add a few drops of methyl red indicator solution and neutralize the solution with just enough NaOH to get a yellow colour. Add about 4 g of mannitol and shake well to dissolve the solid. Add 1ml phenolphthalein indicator solution and titrate with 0.1N NaOH till a permanent pink colour is obtained. The factor (that is, mass of boron in g consumed by each ml of 0.1N NaOH) is calculated as given in **5.4.3**.

5.4.2 *Analysis of Sample*

Take the filtered sample solution (25 ml to 50 ml depending on the boron content) in a beaker, add 3-4 drops of methyl red indicator. Add sodium hydroxide solution cautiously until the yellow colour of the indicator remains. All iron and aluminium will be precipitated at this point.

NOTE — This separation can be replaced by equilibrating the sample solution with a strong cation exchange resin (Amberlite IR – 120) in H^+ form, if the sample solution contains metal ion impurities in small amounts.

Heat the solution gently and allow it to stand for 15 min. Filter off iron and aluminium hydroxide and wash it with hot water. Acidify the filtrate with HCl and heat it at simmering temperature for 10-15 min in a covered beaker. Neutralize the solution and the cover rinsing and washings with alkali to a yellow colour. Now add 3-4 g of mannitol and 1 ml phenolphthalein and titrate with standard alkali to a pink colour. Add 1 g of mannitol, if the pink colour disappears, add more alkali and 1 g mannitol until a permanent end point is obtained. A blank determination is carried out in exactly the same manner.

5.4.3 *Calculations*

a) Factor (g B/ml of 0.1N NaOH) $= \dfrac{10.81 \times A}{61.81 \times V_0}$

where

A = mass of boric acid taken for standardization of alkali, and

V_0 = volume of 0.1N NaOH consumed by (A)g of boric acid.

b) Boron content (g/ml) $= \dfrac{\text{factor} \times V_1}{V_2}$

where

V_1 = titre value (sample – blank), and

V_2 = volume of sample aliquot used for analysis.

5.5 Precision and Accuracy

The accuracy of the titrimetric method is ±5 percent for the determination of 0.02-0.2 mg of boron and for larger amounts of boron the precision is ± 0.1 percent.

6 COLORIMETRIC CURCUMIN METHOD

6.1 Principle

6.1.1 The sample solution is evaporated to dryness in the presence of sodium hydroxide. The curcumin in acetic acid is then added to it. On acidification with sulphuric acid-acetic acid reagent mixture, the curcumin is protonated and reacts with boric acid forming a coloured complex. The unreacted curcumin is then deprotonated with acetone and water mixture.

6.1.2 This method is applicable for samples containing trace quantities of boron.

6.2 Apparatus

6.2.1 *Beakers*, polyethylene.

6.2.2 *Polytetrafluoroethylene (PTFE) Rods*

6.2.3 *Spectrophotometer*, for use at 555 nm, light path 1 cm.

6.3 Reagents

6.3.1 *Curcumin Reagent* — 0.125 percent in glacial acetic acid. Dissolve 0.125 g curcumin in 100 ml glacial acetic acid with stirring. Store in a plastic container.

6.3.2 *Sulphuric Acid-Acetic Acid Reagent (1:1 Mixture)* — Add slowly 50 ml concentrated sulphuric acid to 50 ml of ice cold glacial acetic acid and mix well.

6.3.3 *Sodium Hydroxide Solution* (10 *percent* w/v) — Dissolve 0.143 0 g sodium hydroxide pellets in 100 ml of distilled water in a boron free glass beaker and store it in a plastic container.

6.3.4 *Standard Boron Solution* (1 µg B/ml) — Dissolve 0.143 0 g boric acid in distilled water and make up to 250 ml (that is 100 µg B/ml). Dilute 10 ml of this solution to 1 litre with distilled water (1 µg B/ml).

NOTE — Boric acid looses weight on drying at 105°C. Keep bottle stoppered to prevent entry of moisture.

6.3.5 *Acetone and Water Mixture* (1 : 1) — 50 ml acetone is mixed with 50 ml water.

6.4 Procedure

6.4.1 *Calibration*

Transfer 0.5, 1.0, 1.5 and 2.0 ml of standard boron solution (1 µg B/ml) to a polythene beaker containing 1.0 ml sodium hydroxide. Evaporate gently to dryness on a water bath. Add 3 ml curcumin reagent, warm the beaker to dissolve the residue. Cool to room temperature and add 1.5 ml sulphuric acid-acetic acid mixture and keep for 15 min. Dilute the mixture to 50 ml with acetone-water mixture. Measure the absorbance of the solution and also for blank at 555 nm.

6.4.2 *Analysis of Sample*

Take a suitable aliquot of the filtered sample containing approximately 1.0 µg boron. Equilibrate the solution with 10 ml of Amberlite 1R-120 resin in H^+ form for 1 h to remove cationic interferences from the sample solution. Filter and add sufficient quantity of 10 percent NaOH to make the filtrate alkaline and follow the procedure given in **6.4.1**.

6.5 Calculation

Determine the absorbance of the sample at 555 nm. Compare it with that of standard and calculate the boron content of the sample.

6.6 Precision and Accuracy

The precision of the spectrophotometric method is about ±10 percent for the determination of 1 µg of boron.

7 FLOW INJECTION METHOD

7.1 Principle

7.1.1 Boric acid injected into the flow system reacts with mannitol in the mobile phase and an equivalent amount of H^+ is liberated in the stream. The increase in the conductance of the mobile phase due to the liberated H^+ can be equated to the boron concentration in the sample.

7.1.2 The method is applicable in the range 0.1 – 20 mg/l.

7.2 Interferences

Iron, nickel, copper, cobalt and aluminium do not interfere up to 100 mg/l. Alkali metals can be tolerated up to 1 000 mg/l.

7.3 Apparatus

The flow injection system consist of a pump, an injector valve with a 100 µl sample loop, a conductivity detector and a recorder or an integrator to measure the peak height/peak area.

7.4 Reagents

7.4.1 *Nanopure Water* — Nanopure water having specific resistance 18.3 Ω is to be used for preparing all the solutions. All the reagents used should be of analytical grade.

7.4.2 *Mannitol (0.3 M) Solution* — Dissolve 54.65 g of mannitol in water and then dilute to 1 litre.

7.4.3 *Stock Boric Acid Solution* — Dissolve 0.571 5 g of boric acid in water and then dilute to 100 ml (1 ml = 1 mg of boron).

7.4.4 *Standard Boric Acid Solutions* — Prepare a series of working standards 0.1 - 20 µg/ml by apparopriate dilution of stock boric acid solution with water.

7.4.5 *Strong Anion Exchange Resin in Chloride Form*

7.4.6 Silver columns/cartridges having a capacity of 2 - 25 meq/column on a water swollen basis.

7.5 Procedure

7.5.1 For the analysis of pure boric acid, the aqueous solution can be directly injected into the flow injection system and the conductance/peak height is to be measured.

7.5.2 *Analysis of Samples*

For the analysis of samples, a two step pretreatment procedure is essential.

> *Step* 1 — Equilibrate 10 ml of sample solution with a known weight of the anion exchange resin in chloride form for 1 h, so that all the sample anions are converted to chloride. Filter the solution through a medium textured filter paper. Give four to five washings to the resin with water and make up the final volume to 25 ml.
>
> *Step* 2 — Wash the silver guard column with 8-10 ml of water at a flow rate of 2 ml/min. Take the solution obtained after treatment as described in step 1 in a 10 ml syringe and pass through the Ag column at a flow rate of 2 ml/min. Discard the first 4 ml and collect the rest in a clean and dry beaker. Inject 1ml of this solution into the flow injection system for boron analysis.

7.6 Calibration

Analyze the blank and each of the prepared working standards in accordance with **7.1**. Prepare the calibration curve for boron by plotting the peak height or the peak area *versus* the concentration of boron.

7.7 Calculation

Determine the peak height of diluted sample from recorder chart and obtain the boron concentration (A) in µg/ml from the calibration curve. The boron concentration (B) in the sample is obtained as:

$$B\ (\ g/ml\ ) = A \times 2.5$$

where 2.5 represents the dilution factor

7.8 Accuracy and Precision

The relative standard deviation of the method is ±0.25 percent for the determination of boron at a concentration level of 10 mg/ml.

8 ION CHROMATOGRAPHIC METHOD

8.1 Principle

8.1.1 Boric acid converted to its more acidic complex by reacting with mannitol or sorbitol, is separated on anion exchange column and detected by conductivity detector.

8.1.2 The method is applicable in the range 1 - 200 µ mol/dm^3.

8.2 Interferences

166 µ mol/dm^3 of fluoride, 2 m mol/dm^3 of acetate and 56 µ mol/dm^3 of chloride do not interfere in boron analysis.

8.3 Apparatus

8.3.1 *Chromatographic System* — The system consists of a pump for delivering the eluent, an injector valve with a 100 µl sample loop, a conductivity detector and a recorder or an integrator to measure the area under the chromatogram.

8.3.2 *Guard Column* — A short length column of similar material to the separator column and placed before the separator column is used to protect the main column from being contaminated by particulates and the irreversibly adsorbing constituents.

8.3.3 *Separator Column* — A styrene-divinylbenzene based low capacity pellicullar anion exchange column (Dionex Ionpac As-11SC 4.6 × 250 nm analytical column equipped with a Dionex Ionpac AG11SC, 4.6 × 50 nm guard column).

8.3.4 *Suppressor Column* — A column or membrane is needed that is a high capacity cation exchanger and is capable of converting the eluent and separated anions to their respective acid forms.

8.4 Reagents

8.4.1 *Quality of Reagents* — Analytical grade chemicals and deionized water are to be used in all the preparations.

8.4.2 *Eluent* — Dissolve 54.65 g of mannitol (0.3 M) in water. Add 0.26 ml of 50 percent NaOH and dilute to one litre with water.

8.4.3 *Sulphuric Acid Solution* — Add 2.2 ml of sulphuric acid to 500 ml of water and finally make up to 4 litre to give dilute H_2SO_4 of 1 - 2M.

8.4.4 *Boric Acid Solution* (1ml = 1mg B) — Dissolve 0.571 5 g of boric acid in water and dilute to 100 ml with water and store in polypropylene bottle as stock solution.

Prepare a series of boric acid standards 1 - 20 µ mol/dm^3 by appropriate dilution of the stock boric acid solution. Store in polypropylene bottles.

8.5 Procedure

Equilibrate the system by pumping the eluent through the column until a stable baseline is obtained, after about 30 min. The detector ranges are variable, normal operating ranges are from 3 to 100 µ S/cm, full scale. The range setting required for analysis depends on the concentration of the borate in the sample and should be chosen accordingly. Load the sample loop by injecting about 1 ml of sample into the injection port using a 5 ml plastic syringe. Inject the sample onto the column and record the ion chromatogram.

8.6 Preparation of Calibration Curves

Analyze the blank and each of the prepared working standards in accordance with 7. Prepare the calibration curves for borate by plotting the peak height or the peak area *versus* the concentration of the anion.

8.7 Calculations

Determine the peak height for the sample and obtain the borate concentration in µ mol/dm^3 from the calibration curves.

8.8 Precision and Accuracy

Within the calibration range the precision is better than 5 percent.

9 INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRIC (ICPAES) METHOD

9.1 Principle

The ICPAES technique is based on the excitation of atoms by an argon plasma generated by the application of high frequency RF current. The sample is introduced into the argon plasma through a nebulizer. The emission spectra of the atom is measured using a suitable grating and photomultiplier system. The ICPAES technique is suitable for the analysis of boron in different types of water samples like natural water, potable water, domestic and industrial waste waters. Detection limits of about 100 ppb can be achieved using the wavelength of 208.959 nm and that of about 10 ppb can be acheived using the wavelength of 249.773 nm.

9.2 Apparatus

Inductively coupled Plasma Atomic Emission

Spectrometer (ICPAES). Either sequential or a polychromator system may be employed. Instrument setting as recommended by the manufacturer shall be followed.

9.3 Reagents

9.3.1 *Concentrated Nitric Acid* — conforming to IS 264.

9.3.2 *Stock Boron Solution A (1 000 µg/ml)* — Weigh 0.572 0 g of H_3BO_3 after drying it at 110°C for 1 h and cooling in a desiccator for 1/2 h. Dissolve it in 5 ml of nitric acid and make up to 100 ml.

9.3.3 *Stock Boron Solution B (100 µg/ml)* — Dilute 10 ml of solution A and make up to 100 ml with deionized water.

9.3.4 *Stock Boron Solution C (10 µg/ml)* — Dilute 10 ml of solution B to 100 ml with deionized water.

9.4 Procedure

9.4.1 Follow the procedures as described in manufacturer's instrument operating manual.

9.4.2 *Standardization*

Standardize the instrument and obtain a wavelength profile using a solution of 10 µg/ml of boron solution (*see* **9.3.4**). Using the 10 µg/ml boron solution (**9.3.4**) and demineralized water as high and low standards obtain a calibration curve. Check the reproducibility of the data by repeatedly aspirating a solution of an intermediate concentration level (5 µg/ml). Choose a wavelength of 249.773 nm for the analysis at low concentrations of boron and 208.959 nm for high concentrations. Prepare a test standard of 1 ppm of boron by diluting 10 ml of solution C to 100 ml with deionized water. Use this standard for calculation of precision over short and long periods. Use freshly prepared solutions for all the experiments.

9.4.3 *Analysis of Sample*

Filter the samples solution through a 0.45 µ filter membrane or medium textured filter paper to avoid clogging or choking of the nebulizer. However in the case of water samples containing high salt content (>10 mg/ml), separate the matrix and inject the sample into the plasma for analysis.

9.5 Calculation

Determine the peak height or peak area of the sample and read the boron concentration from the standard calibration curve.

9.6 Precision and Accuracy

The precision of ICPAES measurement is about 2 - 3 relative standard deviation (RSD).

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
In personal capacity *(40/14, C. R. Park, New Delhi 110019)*	Prof Dilip Biswas (***Chairman***)
Bhabha Atomic Research Centre, Mumbai	Dr V. D. Puranik Dr T. N. Mahadeven (*Alternate*)
Bharat Heavy Electricals Limited, Hardwar	Dr N. G. Shrivastava
Cement Manufacturers' Association, New Delhi	Dr K. C. Narang
Central Fuel Research Institute, Dhanbad	Dr Gulab Singh Dr. L. C. Ram (*Alternate*)
Central Leather Research Institute, Chennai	Dr S. Rajamani
Central Mining Research Institute, Dhanbad	Dr B. K. Tewary
Central Pollution Control Board, New Delhi	Dr S. D. Makhijani Dr C. S. Sharma (*Alternate* I) Dr S. K. Tyagi (*Alternate* II)
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Department of Science and Technology (TIFAC), New Delhi	Dr P. Sanjeeva Rao Dr G. Srinivasan (*Alternate*)
Delhi College of Engineering, Delhi	Shri Rakesh Mehrotra Shri V. K. Minocha (*Alternate* I) Dr (Shrimati) A. Mandal (*Alternate* II)
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Indian Institute of Packaging, Mumbai	Shri S. C. Adak
Indian Oil Corporation Limited, Faridabad	Representative

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Member Secretary
SHRI N. K. PAL
Director (Chemical), BIS